DESIGN AND FABRICATION OF APPARATUS FOR USE IN INVESTIGATION OF METALLIC CRYSTAL FORMATION ON GLASS BACKING

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and

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by

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PREFACE

The project described in this paper was undertaken during the 1955-56 academic year at the U. S. Naval Postgraduate School, Monterey, California.

The purpose of the project was to design and fabricate equipment which could be used to investigate the nature of the formation of metallic crystals on glass backing. To date there is little experimental evidence which illustrates in a clear manner the way in which metallic crystals form or nucleate on glass backing. It is hoped that future researchers will be able to use the equipment to investigate matters dealing with this problem.

The writers wish to express their appreciation for the aid and advice given by Professor Eugene C. Crittenden of the Physics Department of the U. S. Naval Postgraduate School.

The writers also wish to thank Mr. Milton K. Andrews and Chief Petty Officer Robert C. Moeller for their assistance in obtaining materials and tools and for their advice on fabrication techniques.

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TABLE OF SYMBOLS AND ABBREVIATIONS

(Listed in the order of their use in the text)

o	-Degrees Centigrade
IR	-Current Multiplied by Resistance
G.E.	-General Electric
emf	-Electromotive Force
AC	-Alternating Current

CHAPTER I

INTRODUCTION

With the development of nuclear power, the problems of radiation damage to metals has become exceedingly important. One method of investigation of radiation damage is to observe the effect of radiation upon a thin film of metal on some suitable backing, such as glass. In order to understand the nature of damage to thin films it is desirable to know something about the inherent defects which may be created in the film during its formation by evaporation. Hence, we must go one step further in our search and seek some information concerning the manner of formation of the original crystals which make up the film.

Preliminary work done by Dr. Eugene C. Crittenden, Jr. of the U. S. Naval Postgraduate School has shown that it is possible to form crystal nuclei by evaporation of certain metals, namely; zinc, cadmium, and mercury, on a glass backing cooled to temperatures in the region of -120° Centigrade. It is then possible to raise the temperature of the backing to a higher value such as 0° Centigrade and bombard the original crystal nuclei with metallic vapor and grow the nuclei to a size such that they may be observed and counted under an ordinary microscope. During this bombardment at the higher temperature no new nuclei are formed. This then provides us with a method for studying the statistical nature of the formation of the original crystal nuclei.

Since it is desirable to form the crystal nuclei under the most ideal conditions so far as purity is concerned, it is necessary that the evaporation take place in an evacuated chamber. In addition, some method of measuring the rate of vaporization of the metal and a means for controlling exposure times are basic requirements.

It was decided to assemble the data collecting portion of the system in a glass cylinder placed between two metal plates. To the lower plate, called the base plate, the vacuum producing system was attached. Two beakers were vacuum sealed into the top plate and a furnace and a shutter device were arranged so that the metal would be evaporated onto the bottoms of the beakers. One beaker was to be used to receive the specimen to be studied, the other beaker was provided with electrical contacts so that the rate of deposit could be evaluated.

The construction of the apparatus then resolved itself into two major divisions: the fabrication and assembly of the vacuum system; and the design, fabrication and assembly of mechanisms to control the evaporation and to measure its intensity.

The remainder of this paper is a description of the equipment and a discussion of its functioning.

Included in the Appendices are suggested techniques and procedures which the writers used in some portions of this work and which they believe will be helpful and time saving to anyone using this particular equipment or any of similar design.

CHAPTER II

THE VACUUM SYSTEM

The vacuum system was designed and installed to remain a permanent part of the research equipment of the Physics Department at the U. S. Naval Postgraduate School. Because of this, a detailed description of the system follows. Reasons for particular design features are also included so that those who follow may have a thorough understanding of why a given part was installed.

The system is divided into two parts for the purpose of discussion: they are the vacuum producing system and the working space. The dividing line between these two sections is a three-quarter inch general purpose steel base plate upon which a variety of experimental systems requiring a vacuum can be installed.

The vacuum producing apparatus consists of a conventional fore pump and diffusion pump. The fore pump is a Kinney compound vacuum pump, Model KC 15 with a theoretical displacement of 7.2 liters per second. The diffusion pump is a four inch fractionating type pump, manufactured by the Consolidated Vacuum Corporation. It has a theoretical displacement of 300 liters per second. However, when the pump is installed the displacement probably lies between 50 and 100 liters per second. The connections between the diffusion pump and the fore pump consists of two short sections of two inch outside diameter steel pipe, two cast steel elbows, a one foot section of flexible steel sylphon bellows pipe, a cast steel adaptor to fit the pipe to the fore pump, a one and one-half inch brass vacuum valve, and two steel

flange adaptors. The flexible steel pipe, vacuum valve and flange adaptors are classed as salvagable, and joints between them and the other parts are sealed with glyptal. Joints between sections of steel pipe are silver soldered. As seen in Figure 1, a flanged adaptor, a 90° cast steel elbow, and the flexible steel pipe form a flanged end section, separating the fore pump from the diffusion pump. This section may be removed to separate the fore pump from the diffusion pump without destroying the glyptal seals.

Soldered into the elbows are three monel metal one-half inch outside diameter tubes. The tube in the elbow nearest the fore pump may be used to attach a standby fore pump. To one of the two tubes in the elbow nearest the diffusion pump is attached a Stokes McLeod gage (Flosdorf modification) for measuring pressure at the fore vacuum end of the diffusion pump. To the other tube is attached a Cenco, "O" ring tube fitting vacuum connector, into which is inserted a Pirani tube. The Pirani circuit is used to detect leaks and is described in Appendix I.

Bolted to the top of the diffusion pump is an aluminum adaptor plate which attaches to a four inch quarter-swing vacuum valve manufactured by the Consolidated Vacuum Corporation. Above this valve is the general purpose base plate.

A sheet aluminum vapor baffle system is supported by the aluminum adaptor plate. The baffles prevent oil vapor from the diffusion pump from entering the working space by deflecting the vapor particles downward.

The system is equipped with three vacuum connectors for the insertion of measuring instruments. One adaptor is located in the aluminum plate separating the quarter-swing valve from the diffusion pump, the other two connectors protruded from the bottom of the base plate.

Various safety interlocks are built into the system. Figure 2 shows a circuit diagram of these interlocks. The interlock circuit protects the system as follows:

a. Cuts off diffusion pump if ion gage reads above 10^{-3} millimeters of mercury.

b. Prevents diffusion pump from starting following a temporary power failure.

c. Cuts off diffusion pump through the thermal switch located near the fore pump end outlet should the pump become overheated.

An alarm lamp is illuminated when the diffusion pump cuts out.

The assembled vacuum system is shown in Figure 1.

CHAPTER III

THE WORKING SPACE

The working space can be varied to suit the need of the project. For the project of crystal nucleation, the working space consists of a 14 inch diameter glass cylinder, approximately 17 inches high. The cylinder is covered by a 20 inch diameter aluminum plate, one inch in thickness.

1. The Base Plate.

Thirteen electrical leads are provided in the base plate and they have been designed to facilitate leak detecting. Figure 3 is a drawing of an electrical lead. The large head of the lead is internal to the working space and the shaft is external. The lead is placed in an oversized hole for insulation purposes and a neoprene gasket smaller than the distance across the flats is used to seal the lead. This provides a shadow zone so that evaporated metal will not short out the lead. The small hole in the shaft provides a means for forcing compressed air or illuminating gas into the hole from the shaft end. If a leak is present around the neoprene gasket it will be indicated on the ion gage or Pirani gage.

The base plate is 20 inches in diameter and will accomodate cylinders or bell jars up to 18 inches in diameter. If a cylinder of less than 14 inches diameter is used the electrical leads will be excluded from the evacuated space. The base plate is provided with holes to accomodate fittings for three inch inside diameter Corning

glass chemical pipe. There are also 16 $\frac{1}{4}$NCZO tapped holes in the base plate for support rods which may be used to support a variety of apparatus. The pumping orifice is a three inch diameter hole. Figure 4 is a photograph of the base plate showing two supporting rods holding the furnace and also showing a copper shield over the pumping orifice. The copper shield prevents small articles from falling into the diffusion pump.

2. The Top Plate.

The top plate is made of aluminum. It is 20 inches in diameter and it is one inch thick. It supports two beakers, a shutter device, various electrical leads and a charcoal trap. Figure 5 is a photograph of the top plate.

3. The Charcoal Trap.

The charcoal trap consists of a helix of $\frac{1}{2}$ inch copper refrigeration tubing surrounded by a cylindrical copper can, open at the top and bottom. Supported between the coils of the copper tubing is a tungsten wire which is insulated with porcelain fishspine beads. Concentric with and interior to the tubing and tungsten wire is a cylindrical copper screen. Fourteen mesh activated charcoal is packed between the screen and the can.

The tungsten wire acts as a heater coil to degas the charcoal while pumping down the system. The heater is connected through a ten volt, 12 ampere filament transformer and a variac to a 110 volt AC line. During the pumpdown operation, 120 watts can be applied to the heater, giving a temperature of approximately 200° Centigrade in the

charcoal, as measured with a chromel p-alumel thermocouple. The power may be doubled for a period of approximately twelve hours. Application of increased power for longer than this period may burn out the heater wire.

The trap is provided with a thermally insulated reservoir located above the top plate. In the final stages of obtaining a vacuum, the coils of the trap can be filled with liquid nitrogen from this reservoir. With the charcoal trap acting as a pump, a vacuum of 4×10^{-6} millimeters of mercury can be sustained.

Figure 6 is a cross section drawing of the trap and its insulating connections.

4. The Furnace.

The metal to be evaporated is held in a conically coiled tungsten wire furnace coated with alundum cement. The furnace is supported from the base plate by steel rods screwed into two of the electrical connectors. Adjustable steel arms attached to the rods hold the furnace. Power is supplied to the furnace by means of a 6-12 volt filament transformer. The shield over the pumping orifice also prevents metal vapor from entering the diffusion pump. See Figure 4.

5. The Beakers.

Two beakers are supported from the top plate, one for the collection of the specimen to be studied, the other for the measurement of the rate at which the metallic vapor strikes the beakers.

The beakers used were 250 cubic centimeter pyrex spoutless beakers. Most of the beakers as received from the supplier are un-

sitable as collecting surfaces and require fire polishing in order to attain the desired surface. The procedure for fire polishing is explained in Appendix II.

The holes through which the beakers were supported were cut at an angle of 11° 6' from the normal to the plate as indicated in Figure 7. This allows the position of the furnace to be adjusted so that it will be equidistant from the bottom of both beakers. The distance from the furnace to the bottoms of the beakers is approximately $6\frac{1}{2}$ inches.

The beakers can be sealed into the top plate by the use of rubber gaskets made by punching $1\frac{1}{2}$ inch holes in 2 3/4 inch rubber discs of 1/16 inch thickness. When the gasket is placed over the end of the beaker and pushed up to the lip there is a quarter inch neck of rubber which presses against the edge of the hole in the top plate. The beaker lips are then covered with a 1/16 inch thick rubber ring and clamped down by steel rings which are secured to the top plate by three screws. To insure that a good seal is made, the tension in the screws must be very nearly equal.

The rate recording beaker is provided with four painted silver electrodes and fitted with a ring containing brass contacts as shown in Figure 8. Leads from this beaker run to the outside through electrical leads in the top plate to a recording device. The beaker is maintained at a constant temperature by being partially filled with liquid nitrogen.

In order to form crystal nuclei on the bottom of the specimen beaker it is necessary to keep the surface of the glass at a low

temperature. This is done by placing a mixture of petroleum ether and liquid nitrogen in the specimen beaker. This mixture is kept in constant agitation by means of a glass stirring rod which is driven by an ordinary household mixer motor. The temperature is measured with a thermocouple. The motor is supported from an adjustable mount which can be seen in Figure 9.

6. The Shutter Device.

In order to control the time during which the collecting surfaces are exposed to the metal source, a shutter device was designed and is shown with the charcoal trap in Figure 8. Figure 10 is a schematic drawing of the various positions of the shutters during a run. The drawing on the left in Figure 10 is the specimen collecting surface and that on the right is the rate recording surface. The dotted line in the drawing is not a shutter but merely a device for opening a shutter that has been closed. It does not cover the aperture.

The sequence of operation of the shutter is as follows:

a. Initially the shutters are closed as shown in position A. This permits degassing by preliminary operation of the zinc source.

b. The recording surface is exposed as shown in position B so that the rate of evaporation could be established. This surface remains exposed throughout the remainder of the run.

c. When the proper rate is established the specimen surface is exposed as in position B.

d. After a predetermined time has elapsed the specimen surface is shielded from the source as shown in C to allow for raising the temperature of the surface.

e. When the specimen surface is isothermal at the new temperature it is exposed as shown in position D, and crystal growth is begun. When crystal growth is to be stopped the furnace power is cut off.

The shutters consist of flat brass plates approximately 1/16 inch thick which are soldered to brass shafts. The shutters are mounted in aluminum housings. On the end of each shaft an arm is fastened to which a 10 ampere fuse wire may be attached. Projecting from the top plate are rods to which the other ends of the fuse wires may be attached. With the fuse wires attached to both the shutter arms and the projecting rods the shutters are in a cocked position and may be tripped electrically by burning out the fuse wire.

In order that no metal from the melting fuse wire may contaminate the surfaces of the beakers, copper shields are mounted on the shutter support frame.

7. The Rate Recording Circuit.

The principle involved in measuring the rate of evaporation of the metallic vapor is to measure the rate of rise in the conductivity of a thin strip of metal being deposited across the painted electrodes on the bottom of the rate recording beaker. During operation this beaker is filled with liquid nitrogen to assure that the metal will stick to the glass and to maintain a constant temperature.

Figure 11 shows the circuit used to record the rate of rise of conductivity. Contacts D_1, R_1, D_2 and R_2 are the painted contacts on the bottom of the beaker. Hanovia silver paste #38 was used to paint these contacts.

A General Electric Self-balancing potentiometer, catalog number 8901490G2, is used to provide a current through the strip of plated metal such that the IR drop across the probes (contacts R_1 and R_2) is equal to the emf set on the Rubicon potentiometer. The varying current from the G. E. Potentiometer is recorded on an Esterline-Angus recorder. This current is proportional to the conductivity of the strip and independent of the contact resistance.

To obtain this circuit the G. E. Potentiometer was modified so that the unknown resistance of the plated strip replaces the standard resistance normally used.

8. The Vacuum.

In testing the system the best vacuum obtained in the working space was 4×10^{-6} millimeters of mercury, with the quarter-swing valve closed and the charcoal trap filled with liquid nitrogen. It is believed that by proper timing of closing the valve and applying the liquid nitrogen this value can be improved. With further regard to improving the vacuum, the beaker seals can be improved by using softer rubber gaskets 3/32 inches thick. This is recommended because the cross section of the beaker is not as circular as desired.

To obtain the best possible vacuum requires approximately 36 hours. About ten minutes after the fore pump has been started the diffusion pump can be turned on. After one-half hour has elapsed the

pressure is sufficiently low to apply power to the trap. Power is applied gradually since the resistance of the tungsten wire increases as its temperature rises. Two hours after starting the fore pump the ionization gage can be turned on and degassed. The pressure will be in the 10^{-4} range. The ion gage is then left on continuously to complete the interlock system.

9. Summary.

The working space is a volume of approximately 80 liters, capable of being isolated from the rest of the system by a quarter-swing valve. It contains the charcoal trap to maintain the vacuum produced, the metallic vapor source and furnace, the collecting and evaporation rate recording surfaces and a shutter device to control exposure time. Operation of this unit is discussed in the following chapter.

CHAPTER IV

TRIAL RUN

Upon completion of construction of the apparatus, it was tested as a unit in a trial run. The mechanical and electrical components functioned as expected. A vacuum of 2×10^{-5} millimeters of mercury was obtained and held when the quarter-swing valve was closed. The shutter firing mechanism opened and closed the shutters with only a slight increase in pressure. Zinc placed in the furnace began to evaporate when the voltage across the filament transformer was 20 volts.

There was, however, one phase which malfunctioned. A copper shield which was thermally connected to the charcoal trap was placed over the furnace so that the source of metal would see a cold surface and evaporated metal would condense only on the intended surfaces. A 7/8 inch hole in the shield collimated the evaporated metal into a beam. In addition to being plated on the bottom of the beaker, which was at the temperature of liquid nitrogen, metal also condensed on the surface of the shield and the charcoal trap. Metal also arrived at the surface of the glass cylinder and condensed; however the glass was slightly above room temperature, having been heated by radiation during outgassing of the trap, and the metal was re-evaporated from the surface. As a result, metal condensed on the side wall of the beaker up as high as the liquid nitrogen level in the beaker. This level was above the band holding the contacts for the painted electrodes. These contacts

shorted out and a very large rate was recorded on the Esterline-Angus recorder.

Figure 12 shows the beaker after it was removed from the system. The extraneous metal is clearly visible in addition to the strip between the painted contacts.

The brief trial run indicates that further shielding is required for the furnace, or in any event, some method to prevent metal from re-evaporating once it has condensed. The latter can be accomplished by coating the surfaces with copper, prior to evaporating the metal to be investigated; and by increasing the size of the shield.

CHAPTER V

CAPABILITIES OF THE SYSTEM

Under optimum conditions a vacuum of approximately 5×10^{-7} millimeters of mercury can be attained. Improvement on this value is limited by the large number of rubber gaskets partially exposed to the high vacuum --29 in all-- and the large quantity of metal parts inside the high vacuum.

The apparatus is able to evaporate metal at various rates and measure and record these rates through a self-balancing circuit. Exposure of the collecting surfaces to the metal source can be controlled by an electrically activated shutter mechanism.

The temperature of the surfaces can be rigidly controlled from outside the system. A stirring mechanism is provided to maintain isothermal conditions.

Cadmium, zinc and mercury are the only known elements which can be studied by the process of crystal nucleation as applied to the device described in this paper.

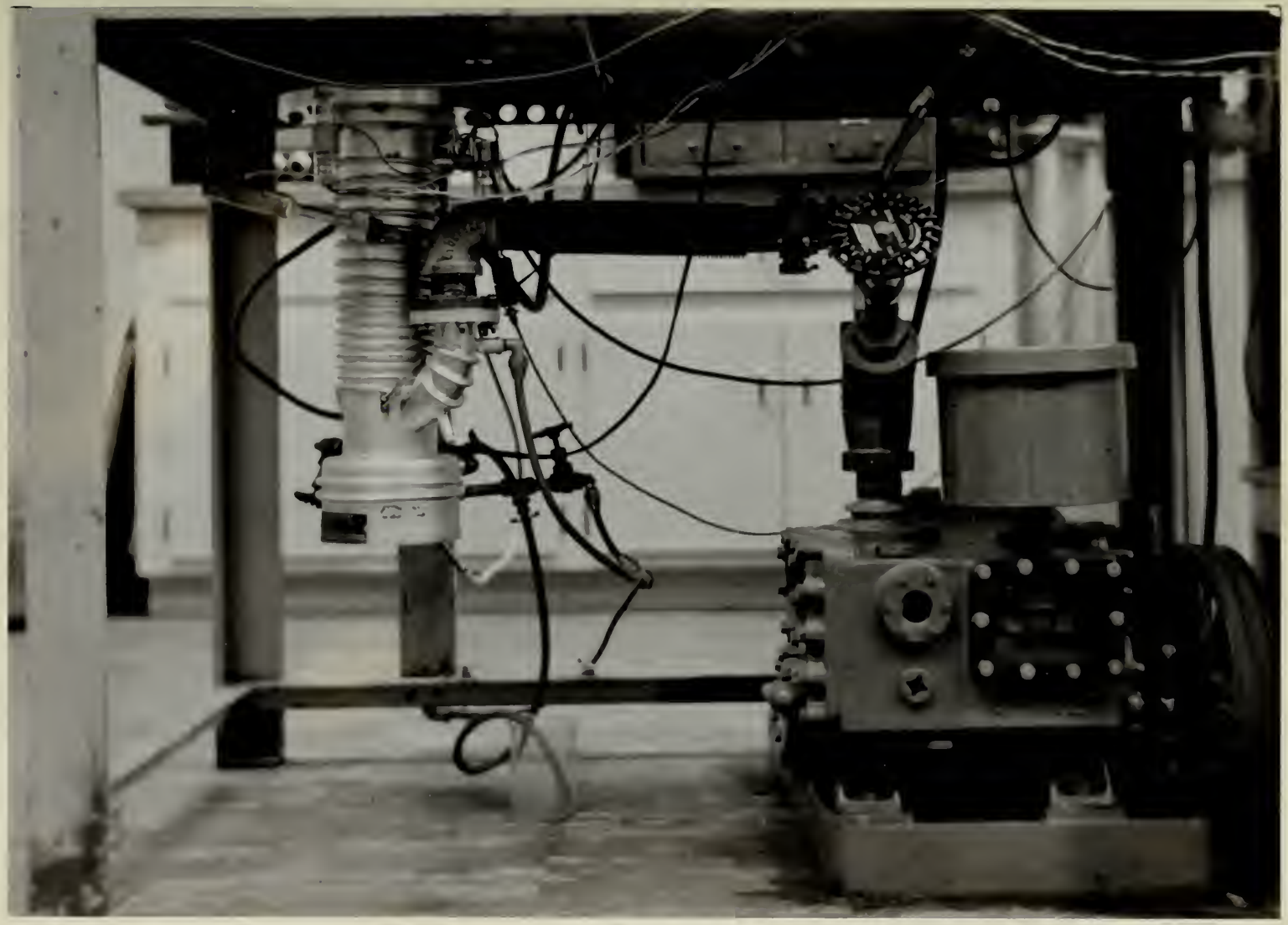

FIGURE 1
THE VACUUM PUMPS

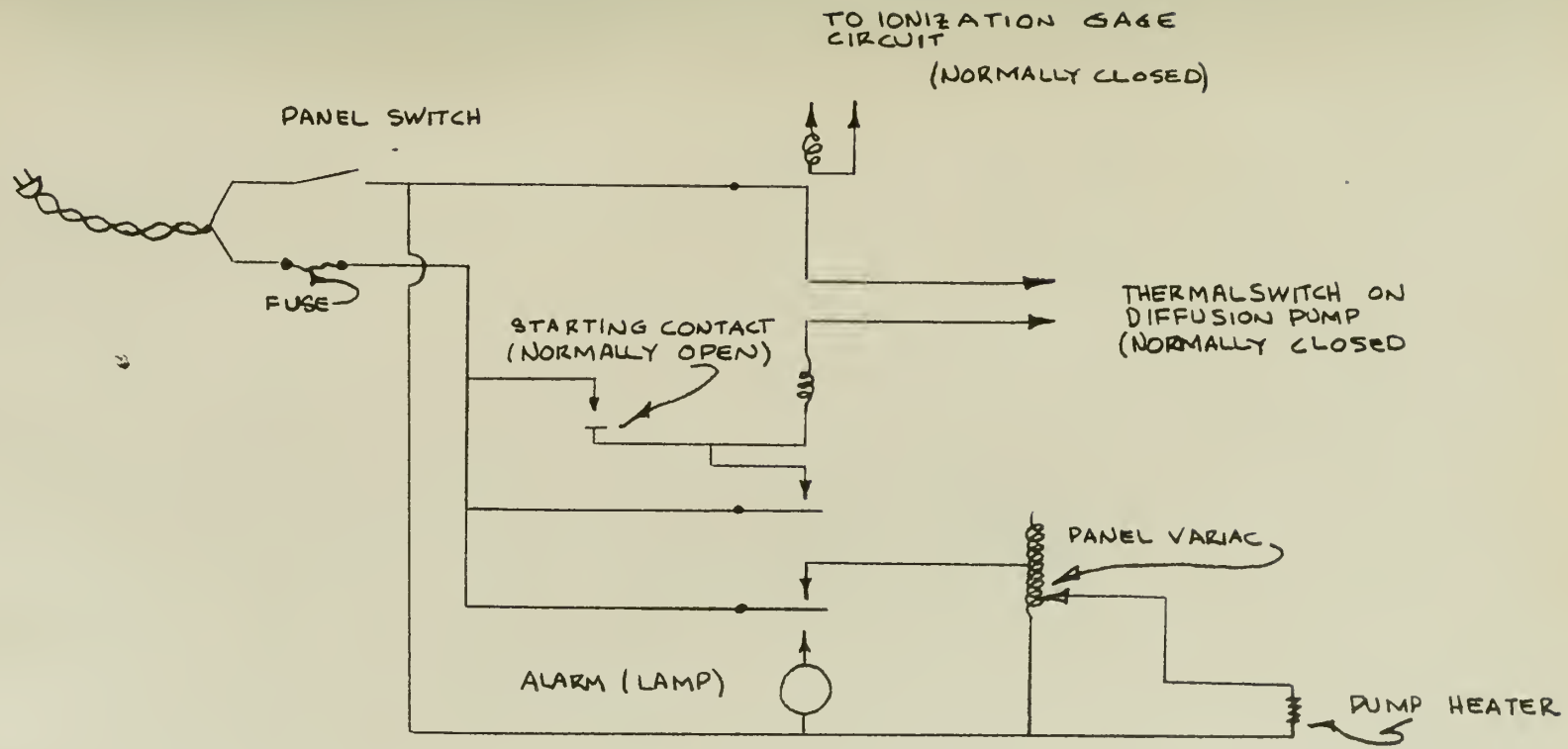

FIGURE 2

PUMP INTERLOCK CIRCUIT

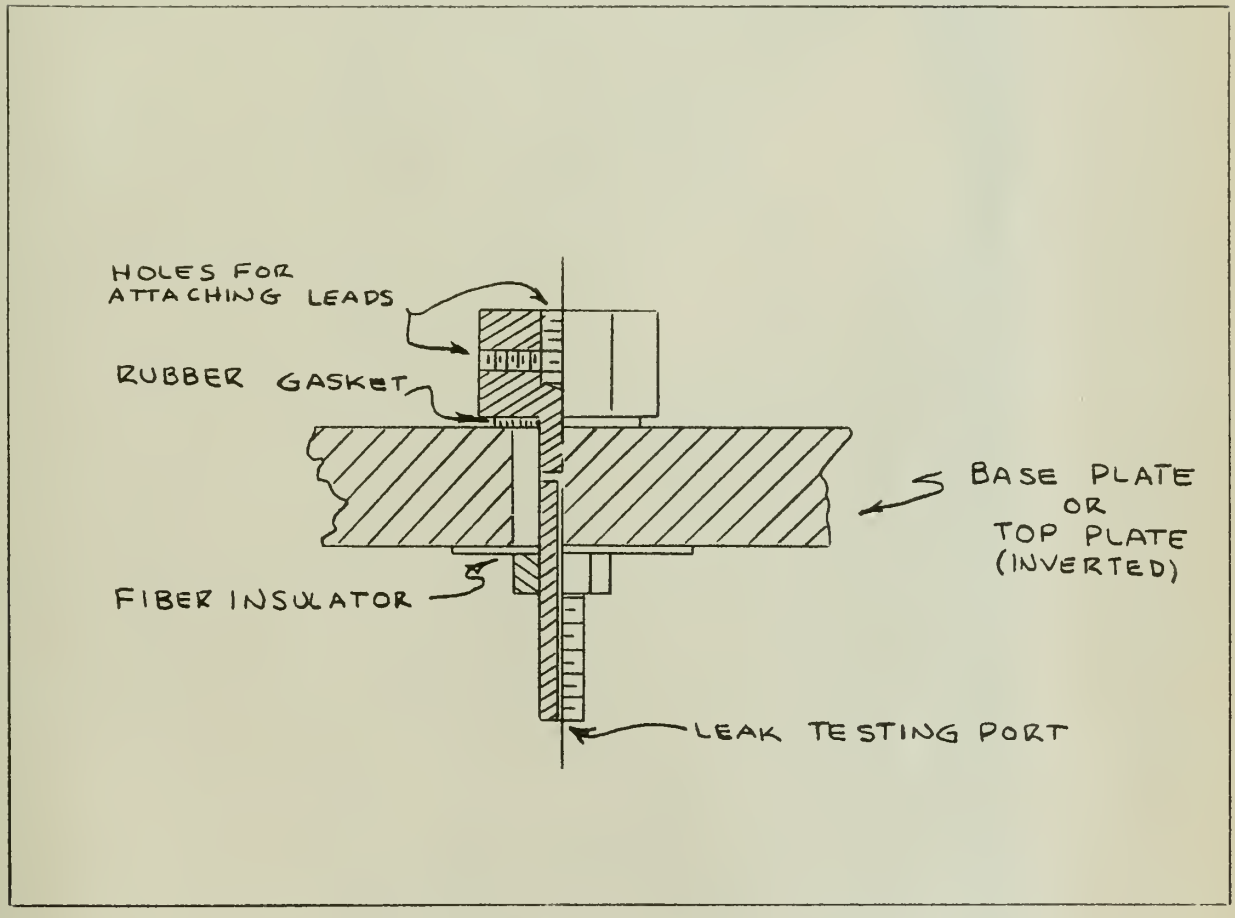

FIGURE 3
ELECTRICAL CONNECTOR

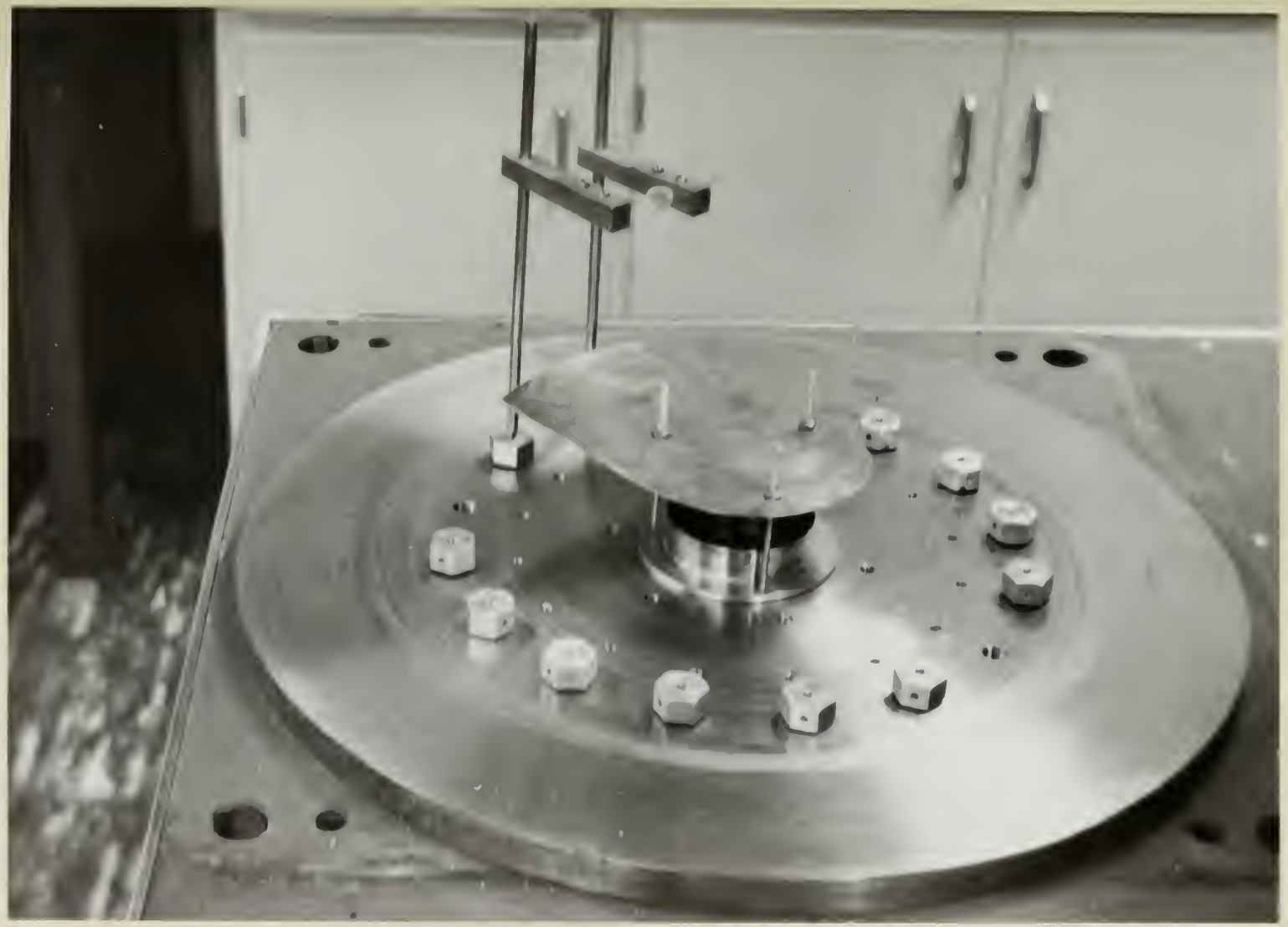

FIGURE 4
THE BASE PLATE

FIGURE 5
THE TOP PLATE

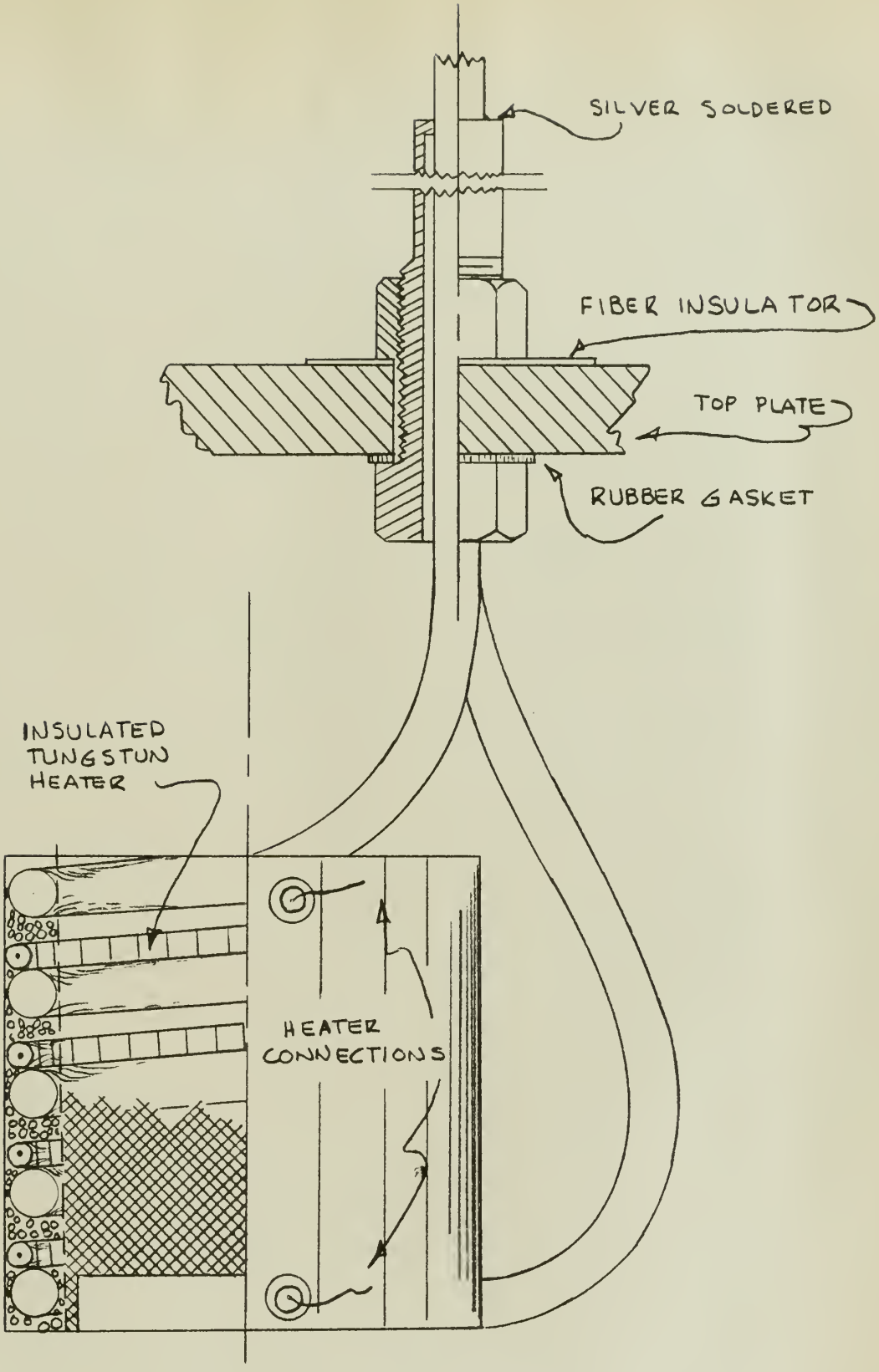

FIGURE 6
CHARCOAL TRAP

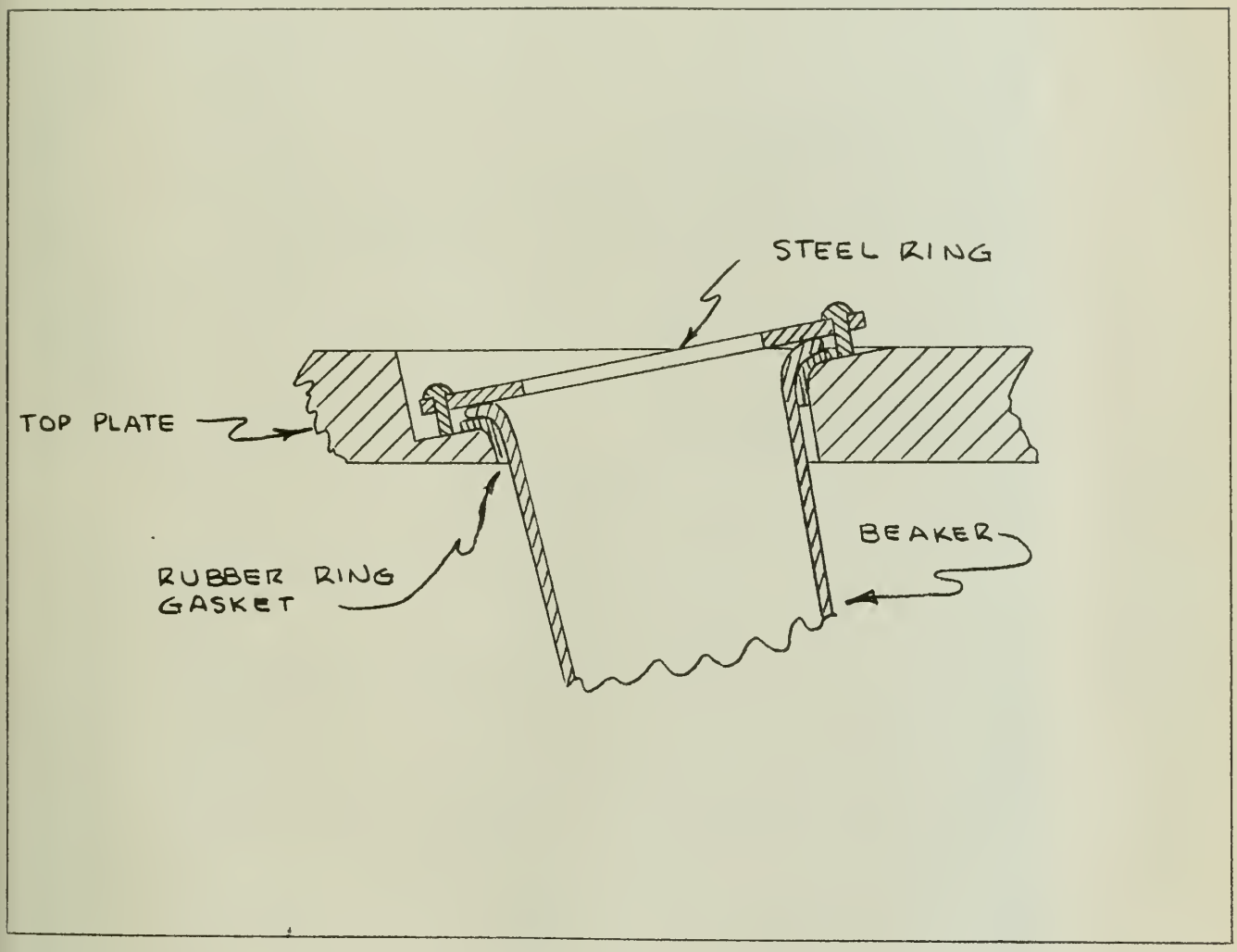

FIGURE 7
BEAKER SEAL

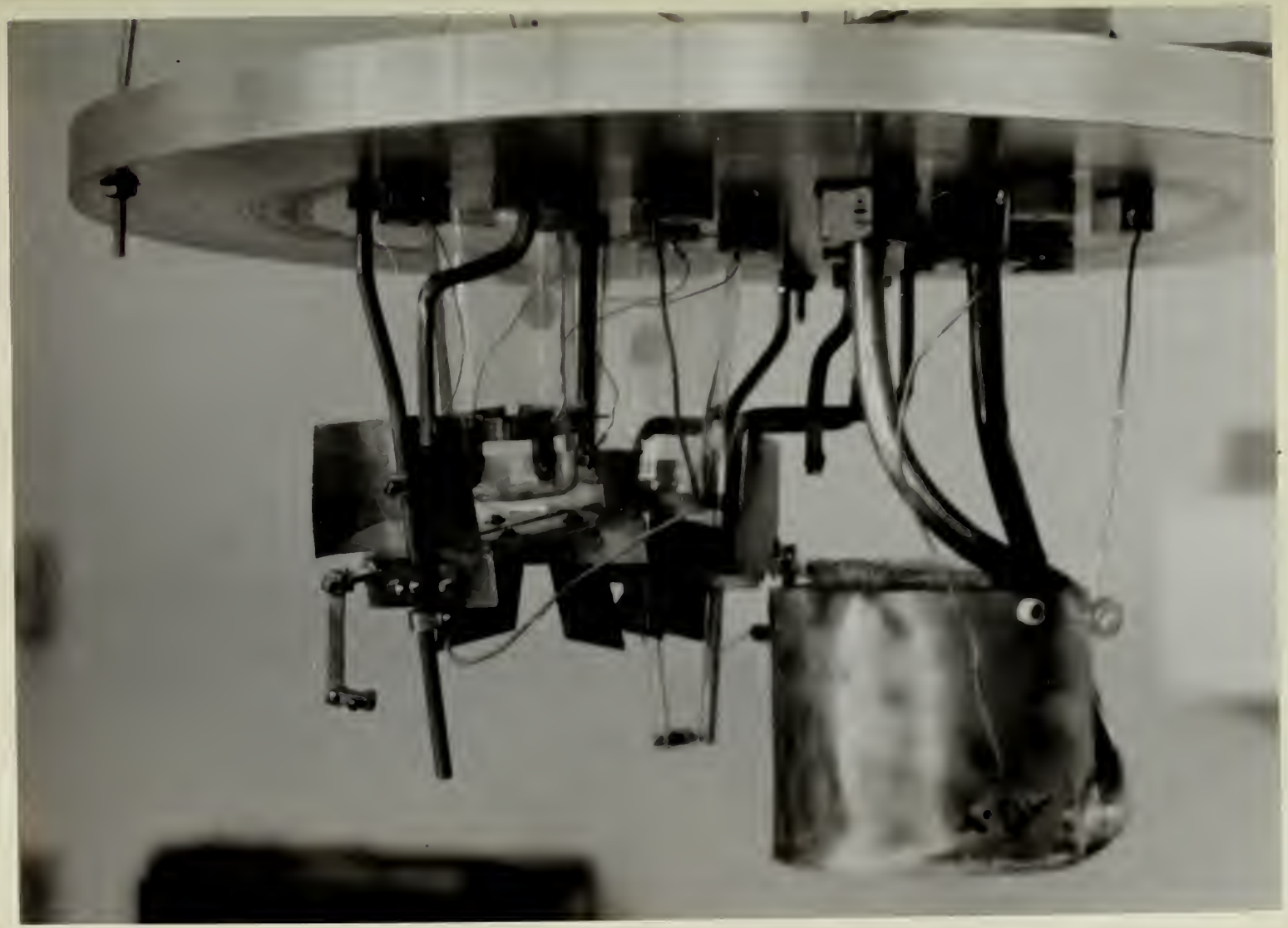

FIGURE 8
CHARCOAL TRAP, BEAKERS, AND SHUTTER DEVICE

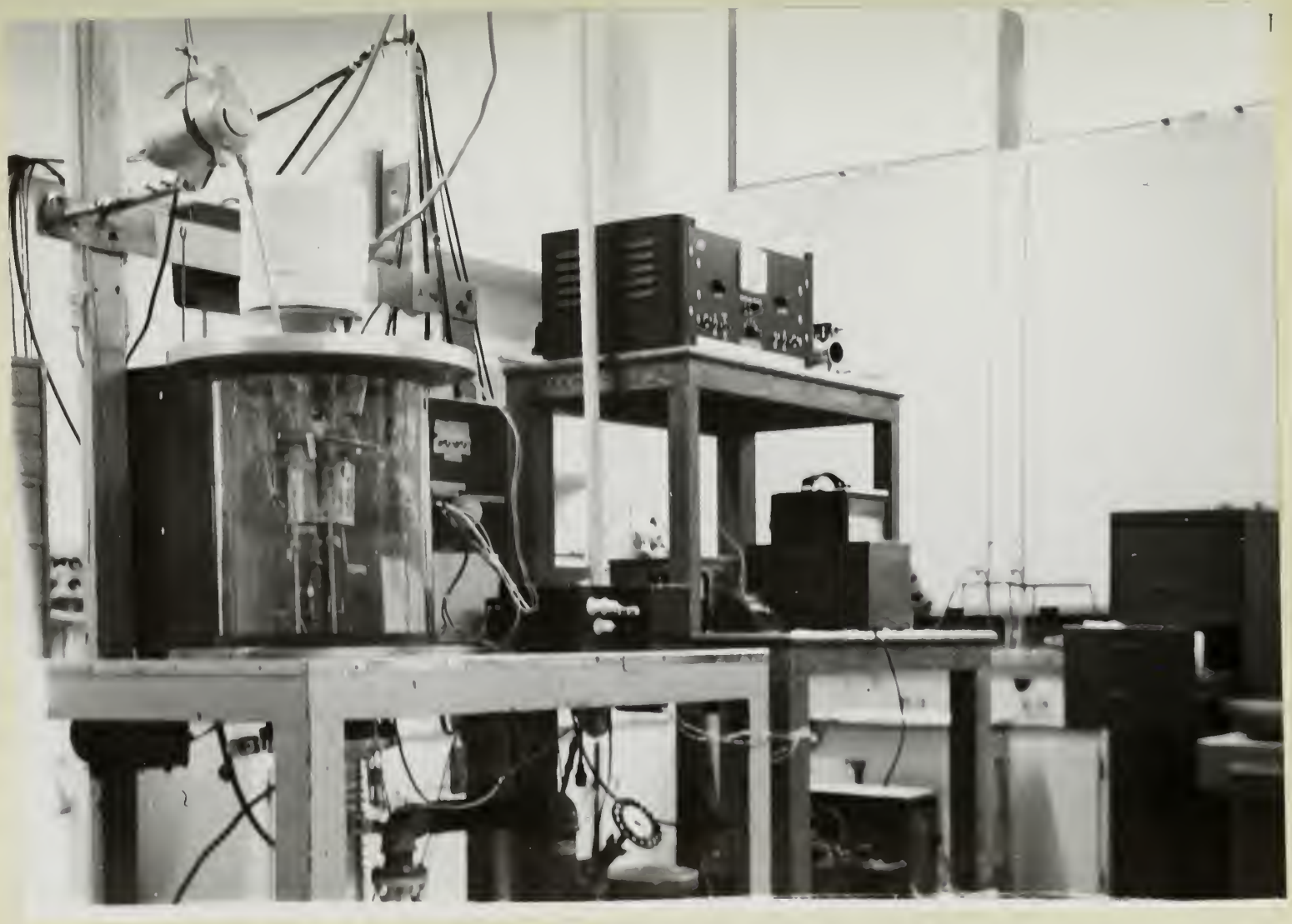

FIGURE 9
OVERALL VIEW OF APPARATUS

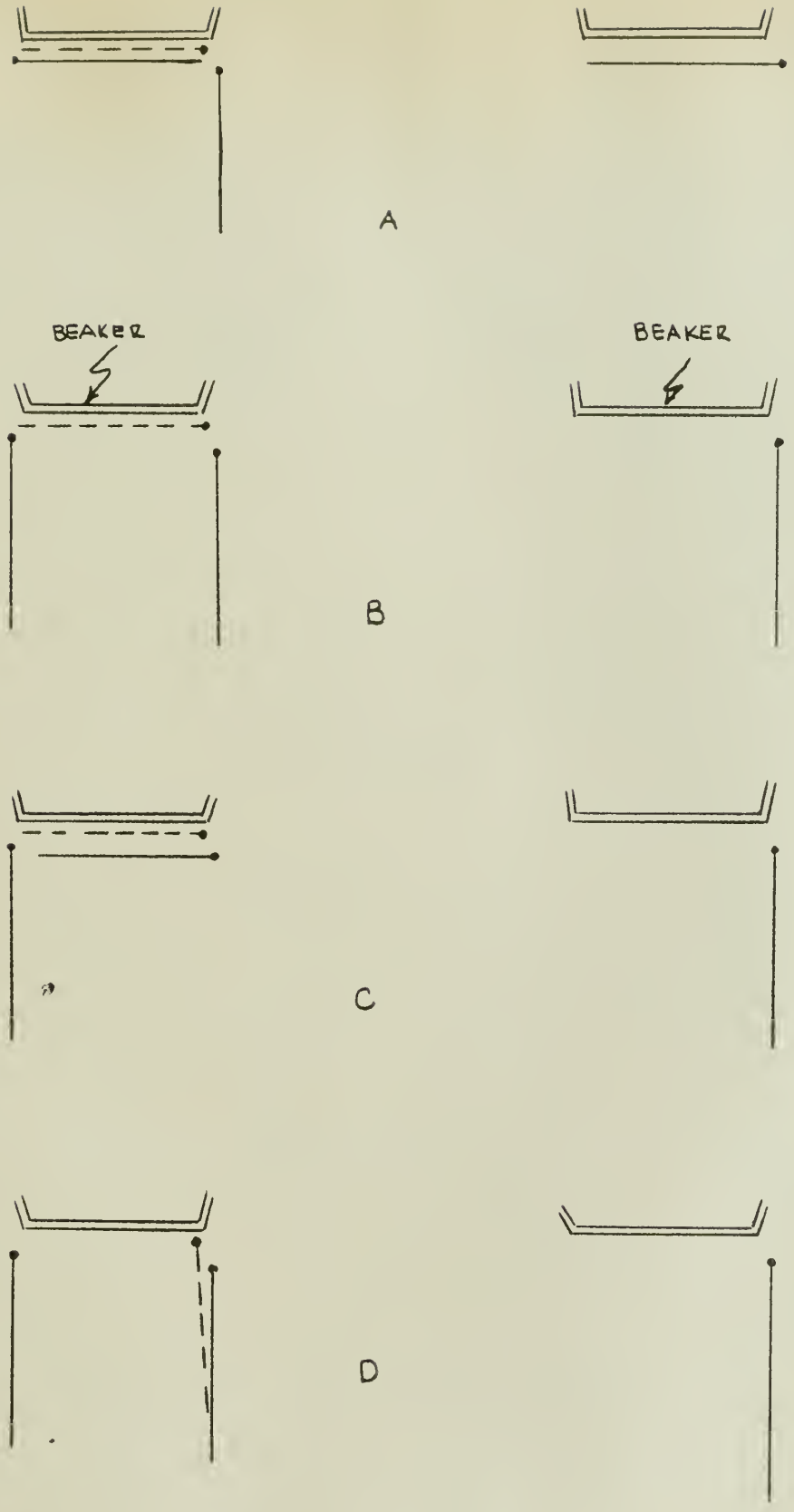

FIGURE 10
SCHEMATIC DIAGRAM OF SHUTTER OPERATION

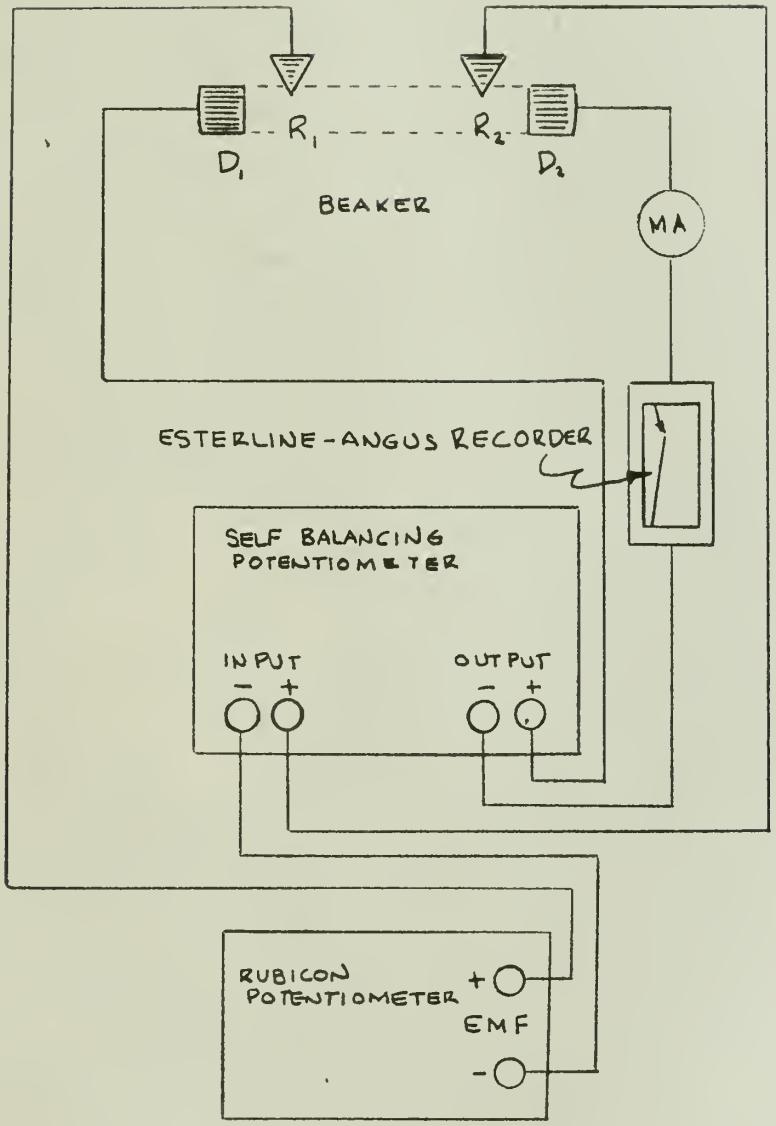

FIGURE 11
CONDUCTIVITY RECORDING CIRCUIT

FIGURE 12
RECORDING SURFACE AFTER TRIAL RUN

APPENDIX I

LEAK DETECTING WITH A PIRANI GAGE SYSTEM

An excellent leak detecting device was installed on the system by using a Pirani gage connected as shown in Figure A-1. The Pirani tube is connected to the system through a connector in the cast steel elbow on the fore pump side of the diffusion pump.

Once the system has been pumped down to a point where leak detecting must begin, the circuit shown is balanced using the highest sensitivity possible on the Ayrton shunt. With the galvanometer needle resting somewhere near the center of the scale, illuminating gas is squirted about suspected leaks. If a leak is found the galvanometer needle will give a wide fluctuation due to the unbalance caused in the system. By using illuminating gas a small amount introduced in the system will cause the conductivity of the Pirani tube to increase greatly compared to the similar introduction of a like amount of air into the system.

Since random fluctuations in the galvanometer needle at high sensitivity may be mistaken for a leak, it is well that experimentation be made with controlled leaks to become familiar with the operation of this system.

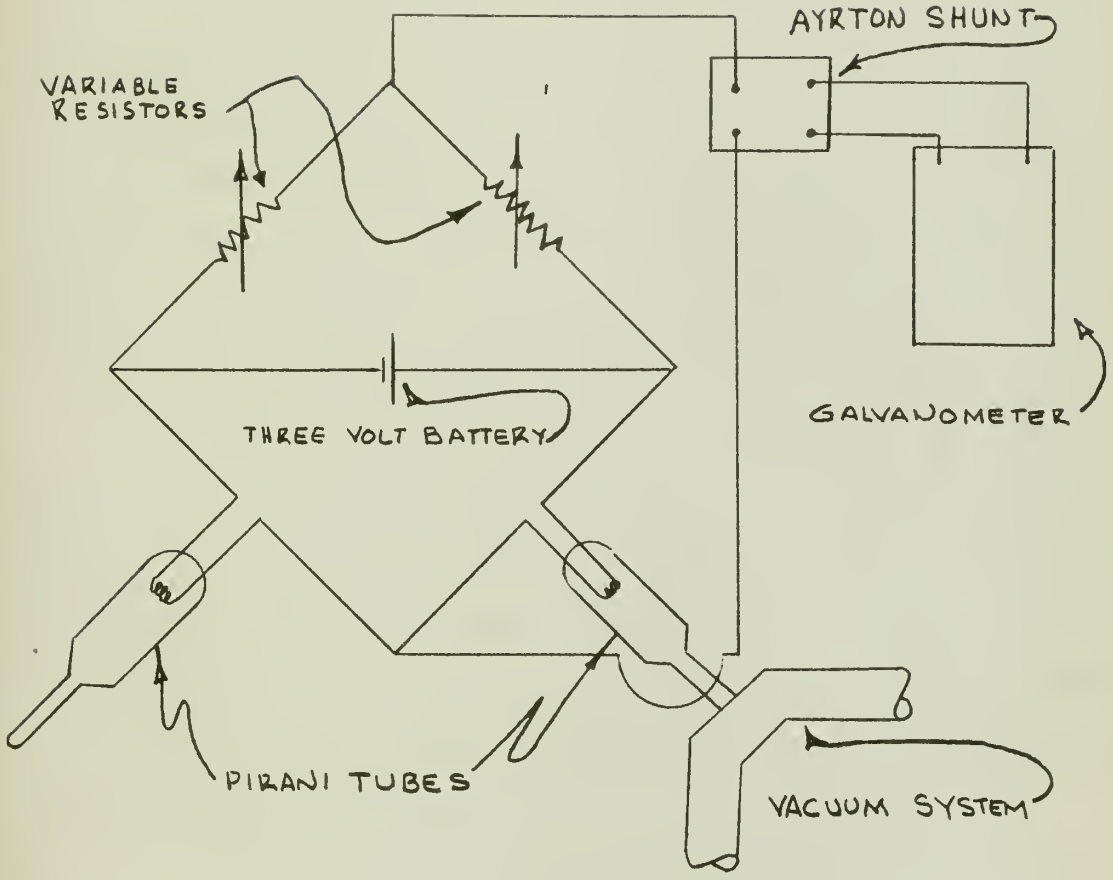

FIGURE AI
PIRANI GAGE CIRCUIT

APPENDIX II

METHOD OF FIRE POLISHING BEAKERS

The beakers which were received from the supplier were found to have very uneven surfaces on the bottom and it was necessary to fire polish them to be sure of local smoothness.

The beaker was mounted on the shaft of an ordinary variable speed AC motor similar to the type used in household batter mixers. A wooden plug was mounted on the shaft and covered with asbestos cloth. The beaker was then pushed down over the plug. A small hole in the plug allowed for the expansion of gas inside the beaker during polishing.

With the beaker mounted on the plug and rotating at low speed the surface being polished is held vertically and a flame is played over the end of the beaker. As the glass warms the speed of rotation is increased and the soft glass will spread evenly across the bottom surface. After the surface becomes even the flame is removed gradually to prevent thermal stresses from developing in the glass. Spinning is continued until the glass hardens.

The flame used in this procedure was produced by illuminating gas and oxygen. Care was taken in adjusting the flame so that no solid particle appeared in the flame. If they appeared they stuck in the soft glass and caused a rough surface.

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